Experiment 8: Spectrophotometric Determination of Iron in Vitamin Tablets

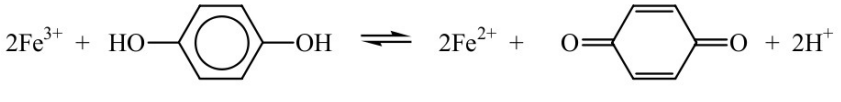
CH2250: Techniques in Laboratory Chemistry, Plymouth State University

Adapted from "22. Spectrophotometric Determination of Iron in Vitamin Tablets," *Experiments To Accompany Exploring Chemical Analysis, 4th Edition*, Daniel C. Harris (2008), available at http://www.whfreeman.com/exploringchem4e. Originally taken from R. C. Atkins, *J. Chem. Ed*, **52**:550 (1975).

Suggested reading for background information: Section Ch 2.10, 4.5-7, 18.1-4 , *Exploring Chemical Analysis, 4th Edition*, Daniel C. Harris (2008).

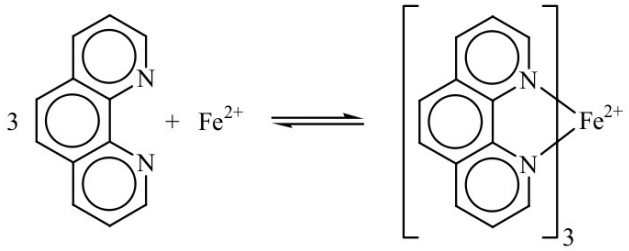
Introduction:

It is often necessary to analyze a specific chemical species in the presence of a number of others. If the species of interest has, or can be made to have, a unique color, spectrophotometric analysis can be quick, easy, and very accurate. In this procedure, iron from a vitamin supplement tablet is dissolved in acid, reduced to Fe^{2+} with hydroquinone, and complexed with o-phenanthroline to form an intensely colored complex (Color Plate 13 in the textbook, between pg 274-275).:



Hydroquinone
FM 110.11

Quinone



o-Phenanthroline
FM 180.21

λ_{max} = 510 nm
Red complex

The absorbance of the iron-o-phenanthroline complex is measured with a spectrophotometer. A series of standards of known concentration of the complex are prepared, and a standard curve of absorbance versus concentration is developed to determine the concentration of the complex in the unknown sample. A standard curve of this nature takes advantage of the linear nature of Beer's law:

$$A = \varepsilon \times c \times l$$

(A is absorbance, ε is molar absorptivity in $M^{-1}cm^{-1}$, c is concentration in M, and *l* is pathlength in cm). By calculation backwards from the standard curve, you will determine the mass of Fe in the vitamin.

Equipment: Read through the procedures and make a list of the equipment you will need.

Safety Considerations: Read through the procedures and note any safety considerations.

Procedure:

The following procedure may require more volumetric flasks than your group has. When you need another flask, pour the solution into a labeled beaker or polyethylene beaker.

A. Preparing the Unknown sample

1. Place one tablet of the iron-containing vitamin and 25 mL of 6 M HCl in a 100 mL beaker, cover with a watchglass, and boil gently (in a fume hood) for 15 min. *While the solution boils, you may move on to the next section, as long as someone is assigned to watch the solutions boil and you do not let it boil too long*.

2. Prepare a piece of filter paper and funnel to do a filtration directly into a 100 mL volumetric flask. Wet the filter paper with a few milliliters of distilled water.Carefully filter the hot solution. Wash the beaker and filter several times with small portions of water to complete a quantitative transfer. Allow the solution to cool to room temperature.
3. Dilute to the mark with distilled water and mix well. *This is your Unknown Digested Solution.* Transfer it to a clean beaker or plastic bottle. Use a permanent marker to label the container.
4. Transfer 5.00 mL of Unknown Digested Solution to a 100 mL volumetric flask. Dilute to the mark with distilled water and mix. *This is your Unknown Stock Solution.* Transfer it to a clean beaker or plastic bottle. Use a permanent marker to label the container.
5. Fill a buret with sodium citrate solution provided by the instructor. Note the initial volume.
6. Pipet 10.00 mL of Unknown Stock Solution into a beaker and measure the pH. Add sodium citrate solution from buret slowly and carefully until a pH of ~3.5 is reached. Note the volume in the buret. This will require 3-10 mL of sodium citrate solution.
7. Transfer 10.00 mL of Unknown Stock Solution to a clean 100-mL volumetric flask. Add the same amount of citrate solution from the buret determined in Step A7. Add about 50 mL of distilled water (*do NOT fill the flask yet!*), stopper the flask, and set it aside for now. *This is your Unknown Sample Solution. Be sure to label the flask*.

B. Preparing Standards

1. Obtain about 40 mL of Fe Standard Solution provided by the instructor (~0.04 mg Fe/mL; note the exact concentration written on the bottle of solution) in a beaker.
2. Pipet 10.00 mL of Fe Standard Solution into a beaker and measure the pH. Add sodium citrate from the buret until a pH of ~3.5 is reached, noting the volume of solution required.
3. Pipet a fresh 10.00-mL aliquot of Fe standard into a 100-mL volumetric flask and add the same amount of citrate solution as required in Step 2. Add about 50 mL of distilled water, stopper the flask, label it, and set it aside. *Do NOT fill the flask yet! This is your Standard 1. Be sure to label the flask.*
4. Repeat Step B2 using 5.00, 2.00, 1.00 and 0.00 mL (a "blank") of Fe Standard Solution. Use sodium citrate solution in proportion to the volume of Fe solution. (If 10 mL of Fe requires 2.0 mL of citrate solution, 5 mL of Fe requires 1.0 mL of citrate solution.) As you prepare each solution, add about 50 mL of distilled water, stopper the flask, label it (Standard 2, Standard 3, etc.), and set it aside.

C. Spectroscopic Measurement

1. To each solution (Unknown Sample from A8 and five standards from B3 and B4) add 2.00 mL of hydroquinone solution and 3.00 mL of o-phenanthroline solution, dilute to the mark with water, and mix well. Wait 10 min before analyzing the solutions.
2. Calibrate a Spec 20 Colorimeter and set its wavelength to 510 nm.
3. Measure the absorbance of each solution at 510 nm using the Spec 20.

D. Disposal of Solutions

1. Dispose of all solutions in the appropriate waste container.

Analysis

1. Calculate the concentration of Fe in all of the standards in the units micrograms per liter (μg/L) . Be sure to use the actual concentration of the Fe Standard Solution (B1) and the correct dilution factors (B3 and B4). *Remember to include a sample calculation in your notebook.*
2. Using Excel, make a graph of absorbance versus concentration (μg/L) of Fe in the standards. Be sure to include the "blank" solution with 0 μg/L of Fe.
3. Using Excel, find the slope, intercept, standard error of linear analysis, and R-squared for the standard curve. *Refer to Lab 8 if you do not remember how to do this.* Report the equation of the line and the two statistical numbers in your notebook. Print a copy of the graph for inclusion in your notebook.
4. Using the calibration curve (i.e., equation of the line from Question 3), find the concentration of Fe in your Unknown Sample Solution (A8) in μg/L.
5. From your answer for Question 4, find the concentration of Fe in your Unknown Stock Solution (A5) in μg/L, then the concentration in your Unknown Digested Solution (A4).
6. Determine the mass of Fe in your Unknown Digested Solution. *Note that this is the mass of Fe in the vitamin as well!*
7. From your answers for Question 1, calculate the molarity (mol/L) of Fe in each Standard solution.
8. Using Beer's law (see Introduction), calculate the molar absorptivity (ε) for each solution and the average molar absorptivity (with standard deviation) overall for $Fe(o\text{-phenanthroline})^{2+}$.

Conclusions

1. Comment on the values of the standard error of linear analysis and R-squared as indicators of the uncertainty in the values calculated for the unknown sample.
2. The vitamin tablets claim to have 15 mg of Fe per tablet. Calculate the percent difference between this theoretical value and your experimental one.
3. Name two potential sources of error in determining the mass of iron in the vitamin tablets. Do you trust that each tablet contains exactly 15 mg of Fe more or less than you trust your own analytical technique?

Homework Problems

The following problems from your book must be completed in your lab notebook (see the Syllabus for other suggested problems): Ch 4: **15**; Ch 18: **3**, **4**, **11**, **15**